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## Treated Textiles and Compositions for Treating Textiles

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This application claims priority to U.S. Patent Application No. 60/504756, filed on September 22, 2003.

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5           Stain release refers generally to the ability of a textile or fabric to release a  
ground in stain, such as an oil-based stain, from the fabric surface. It would be  
beneficial if a textile was capable of exhibiting stain repellency to liquid spills, but also  
function well in stain release. Many prior art treated textiles provide protection in terms  
of repellency, but fail to provide substantial protection as to stain release. A fabric that  
10       could provide repellency to liquids, substantive stain release, and also control the  
growth of microbes, mold, mildew, and the like, would be highly desirable for many  
textile surfaces.

          A recent U.S. Patent application publication 2003/0008585 A1 to Rubin et. al.  
entitled "Treated Textile Fabric" ("Rubin") discloses compositions and a process for  
15       preparing a treated textile fabric. In general, the fabric comprises from about 6 weight  
percent to about 12 weight percent of a fluorochemical textile treating agent, which  
forms a relatively heavy film on the fabric. However, a textile containing such a  
relatively large percentage of textile treating agent may be undesirably stiff or have a  
relatively low degree of hand. Further, such chemicals are costly when applied in such  
20       amounts. Using relatively large amounts of such treating agents upon a fabric surface  
may reduce softness, which is undesirable. Also, such fabrics have poor soil and stain  
release characteristics.

### **Detailed Description of the Invention**

          Surprisingly, it has been discovered that certain compositions are capable of  
25       affording to a textile surface good liquid repellency, while also imparting substantive  
stain release. Furthermore, bacterial growth simultaneously may be controlled by the

5 use of antimicrobial components or agents. A crosslinking component may also be  
employed in the composition, as an optional component. Such treatments provide long  
lasting effects, that is, textiles so treated are in general durable to normal use, such as  
wear and tear. The advantageous properties as described will last even after many  
cleanings and long term uses. Furthermore, it is possible to provide such  
10 advantageous effects without forming a heavy film on such textile articles. Many of the  
applications of the invention (but not all) use less than about 6 weight percent of  
fluorocarbon as a percentage of the total or primary treatment composition.

#### Definitions and Terms

15 "Water repellency" and "oil repellency" are generally defined as the ability of a  
substrate to block water and oil from penetrating into the substrate, respectively. For  
example, the substrate may be a textile substrate which is capable of blocking water  
and oil from penetrating into the fibers of the textile substrate.

"Stain and soil release" generally refers to the degree to which a stained  
substrate approaches its original, unstained appearance as a result of a care procedure.

20 The terms "stain or soil resistant composition or stain or soil resistant treatment"  
as used herein refer to any treatment or composition that imparts stain resistance to  
fibers, particularly polyester or blends.

"Durability" is generally defined as the ability of a substrate to retain an  
acceptable level of a desired function through a reasonable number of cleaning or wear  
25 cycles. More specifically, durability, as described herein, describes a substrate that  
maintains adequate properties of stain resistance, water repellency, oil repellency, and

5 soil release over the life of the product. This substrate may be a textile substrate, such as, for example, a polyester textile fabric, or alternatively may be a carpet, or yet another textile material.

The terms "fluorocarbons," "fluoropolymers," and "fluorochemicals" may be used interchangeably herein and each represents a polymeric material containing at least  
10 one fluorinated segment.

"Hydrophilic" is defined as having a relatively strong affinity for or ability to absorb water.

"Hydrophobic" is defined as lacking affinity for or the ability to absorb water.



5 exchange compounds, inorganic antimicrobial materials, metal based zeolites, metal salts, metal oxides, metal hydroxides, transition metal ions, triclosan, pyrrhione and derivatives, tributyl tin oxide derivatives, 3-iodo-2-propylbutyl carbamate, n-butyl-1, 2 benz-iso thiazoline, 10, 10'- oxybisphenoxiarsine, sodium o-phenylphenate, and others, to name only some of the possible choices that may be employed.

10 In many applications, it will be desirable to employ silver-based ion-exchange compounds, a silver-based zeolite, or a silver-based glass, and any combinations thereof. One silver-based ion exchange material is an antimicrobial silver zirconium phosphate (RC-5000®) available from Milliken & Company, under the tradename ALPHASAN.

15 Generally, such a metal compound may be added in an amount of from about 0.00001 to 10% by total weight of the particular latex composition; or alternatively from about 0.001 to about 5%; or otherwise, from about 0.01 to about 1%; and also from about 0.1 to about 1.0%.

20 Antimicrobial agents such as Ultrafresh NM™ and Ultrafresh DM-50™, DM-25™ (from Thompson Associates), RC-5000™ (from Milliken Chemical), Chitosante™ (VAG Bioscience, Inc. R.O.C., Taiwan), Kathon LM™ (from Rohm and Haas Company), Zinc Omadine (from Arch Chemical), Reputex 20™ (from Avecia), AM 5700™ (Dow Corning), Amical 48™ (Dow Chemical Co.), also may be employed. In many applications, Zinc Omadine or Sodium Omadine are very effective antimicrobial agents.

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5 Inc. and Mitsubishi International Corporation, each of Japan could be employed, as well as others. REPEARL® F-8025, manufactured by Mitsubishi International Corporation may be used as well. Fluoroacrylates and urethane derivatives may be employed. Esters, (meth)acrylic amides oligomers and polymers also may be employed.

10 Optional Generation of a  
Static Dissipative Fabric or Textile

One particular embodiment of the invention may employ materials necessary to make a static dissipative textile having an electrically conductive surface. This may be achieved by first applying a fluorochemical, an antimicrobial agent, (separately or  
15 together, in any order) and optionally a crosslinker or repellent, followed by the application of a static dissipative material. The electrically conductive surface may be achieved by screen printing the fabric with an electrically conductive coating, wherein the conductive coating includes a conducting agent and a binding agent, and optionally a dispersing agent and/or a thickening agent. The fabric may be coated in any pattern  
20 which achieves the desired static dissipative property for the fabric end-use. The fabric may be coated on one or both sides as determined generally by the end-use of the fabric by considering the desired appearance of the coated fabric or the conductive performance of the coated fabric. The resulting electrically conductive fabric may be suitable in end-use applications such as automotive upholstery and other automotive  
25 interior fabrics, such as door panels, armrests, headrests, commercial and/or residential upholstery; cleanroom garments, wipes and/or other cleanroom accessories such as mops, napery, and apparel.



5           In one embodiment of the invention, it may be possible to achieve a composite material, wherein a static dissipative textile may further comprise at least one layer of a second fabric disposed adjacent to the electrically conductive coating. The second fabric may be woven, knitted, or nonwoven fabric. Alternatively, the static dissipative textile may further comprise at least one layer of foam material disposed adjacent to the electrically conductive coating. The composite material may further include one or more  
10 layers of woven, knitted, or nonwoven fabric; one or more layers of film; one or more layers of adhesive; and combinations thereof.

          The composite material may be used, for example, in automobile interiors, such as in automotive upholstery, wherein the upholstery fabric is adhered to a foam backing  
15 through the use of adhesive, heat lamination, or the like. The composite material may be applicable for use in other areas such as, for example, in residential or commercial upholstery or in carpeting.

          It is also an object of the current invention to achieve a method for producing a static dissipative textile having an electrically conductive surface. The method generally  
20 comprises the steps of providing a knitted, woven, or nonwoven fabric, coating one or both sides of the fabric with an electrically conductive coating in a pattern comprised of lines, and drying the fabric. The fabric may then be exposed to one or more mechanical and/or chemical textile finishing processes known to those skilled in the art.

          A static dissipative textile is provided which has relatively permanent anti-static  
25 properties which are achieved at substantially all relative humidities without significantly compromising the textile hand (or feel) of the textile or the surface appearance of the

5 textile. The static dissipative textile generally comprises a fabric coated on at least on side with a pattern of an electrically conductive coating.

### Textile Substrates

Textiles substrates employed in the practice of the invention which are to be treated may be synthetic, natural, and/or blends. They can be woven, knit, carpet or  
10 nonwoven. The composition(s) may be applied to textile substrates by generally known methods such as immersion, foam, spray, exhaustion, and coating. Such compositions can be applied to either side or both sides of the textile substrates. Such compositions could also have one or more components applied to the substrate, followed by other or all components. In addition, such compositions could have one or more components  
15 applied to one side of the substrates, and other or all components applied to either side of the substrates.

The fabric of the current invention can be formed from fibers such as synthetic fibers, natural fibers, or combinations thereof. Synthetic fibers include, for example, polyester, acrylic, polyamide, polyolefin, polyaramid, polyurethane, regenerated  
20 cellulose, and blends thereof. More specifically, polyester includes, for example, polyethylene terephthalate, polytriphenylene terephthalate, polybutylene terephthalate, polylactic acid, and combinations thereof. Polyamide includes, for example, nylon 6, nylon 6,6, and combinations thereof. Polyolefin includes, for example, polypropylene, polyethylene, and combinations thereof. Polyaramid includes, for example, poly-*p*-  
25 phenyleneteraphthalamid (i.e., Kevlar®), poly-*m*-phenyleneteraphthalamid (i.e.,

5 Nomex®), and combinations thereof. Natural fibers include, for example, wool, cotton, flax, and blends thereof.

The fabric can be formed from fibers or yarns of any size, including microdenier fibers and yarns (fibers or yarns having less than one denier per filament).

10 Furthermore, the fabric may be partially or wholly comprised of multi-component or bi-component fibers or yarns which may be splittable along their length by chemical or mechanical action. The fabric may be comprised of fibers such as staple fiber, filament fiber, spun fiber, or combinations thereof.

15 Additional textile treatments can be applied together or separately on either side of textile substrates. Examples include durable press resins and catalysts, sewing lubricants, softeners, antistatic treatments, flame-retardants, and light stabilizers.

#### Test Methods

Liquid or stain resistant properties may be measured using water and oil repellency tests.

20 a) Water Repellency may be tested according to the 3M Water Repellency Test II (May, 1992). The rating scale is 1 – 10, with “1” indicating the poorest degree of repellency (substrates having higher surface energy) and “10” indicating the best degree of repellency (substrates having lower surface energy). The 3M water repellency scale is:

- 1 is 10% IPA, 90% water
- 2 is 20% IPA, 80% water
- 3 is 30% IPA, 70% water

- 5
- 4 is 40% IPA, 60% water
  - 5 is 50% IPA, 50% water
  - 6 is 60% IPA, 40% water
  - 7 is 70% IPA, 30% water
  - 8 is 80% IPA, 20% water
- 10
- 9 is 90% IPA, 10% water
  - 10 is 100% IPA

b) Oil Repellency may be tested according to the AATCC Test Method 118-1983.

The rating scale is 1 – 8, with “1” indicating the poorest degree of repellency (substrates  
15 having higher surface energy) and “8” indicating the best degree of repellency  
(substrates having lower surface energy). The oil repellency scale is:

- 1 is Nujol™ Mineral Oil
  - 2 is 65/35 Nujol/n-hexadecane (by volume)
  - 3 is n-hexadecane
- 20
- 4 is n-tetradecane
  - 5 is n-dodecane
  - 6 is n-decane
  - 7 is n-octane
  - 8 is n-heptane

5 c) Stain release properties may be measured by using a spot cleaning procedure.  
Oily stains such as corn oil and tanning oil were pressed into the textile substrate  
using the staining procedure described in AATCC Test Method 130-1981; as  
modified herein. The stained textile was left at room temperature for 24 hours. A  
piece of paper towel was used to wipe off excess stains at the surface of the  
10 textile. Then 4 drops of fabric cleaner (such as Zout) were gently worked into the  
stained area. The textile was left for 5 minutes at room temperature. Finally the  
stained areas were scrubbed with approximate 4" by 4" polycotton cloths for  
about 40 seconds and then rinsed with generous amount of warm water. Excess  
water was blotted off with a paper towel. After the cleaned textile was air dried at  
15 room temperature, stain release performance was rated against the rating replica  
of AATCC 130-1981 with a rating scale from 1 to 5 with 5 to be the best.  
Generally a rating of 3.5 and above is considered to have good soil/stain release  
property.

#### Antimicrobial Testing

20 Antimicrobial properties (bacteria and fungi) may be tested using modified  
AATCC Method 147, also known as the Parallel Streak Method, as further defined  
below.

In the Parallel Streak Method, an agar surface is inoculated, thereby making it  
easier to distinguish between the test organism and contaminant organisms that may be  
25 present on the unsterilized specimen. The Parallel Streak Method has proven effective

5 in providing evidence of antibacterial activity against Gram positive and Gram negative bacteria.

A Zone of Inhibition (millimeters) and growth under the fabric were used to gauge antimicrobial properties of the textile substrates. Generally no growth under the substrate and/or a clear zone of inhibition (ZOI) around the substrates indicates good antimicrobial properties.

#### Zone of Inhibition

Migration of the antimicrobial was assessed with the Zone of Inhibition assay. Petri plates containing Tryptic Soy Agar were inoculated with 0.5ml of a diluted overnight culture approximately  $5 \times 10^5$  cells/ml in Na/K phosphate buffer of the test microbe. Samples were tested against *Klebsiella pneumoniae* ATCC No. 4362 and *Staphylococcus aureus* ATCC No. 6538, and *A. niger* (a fungus). A sample approximately 1 x 1 inch is placed in the center of the plate. The agar plate was incubated for 24 hours at 35°C. The final data is the average of the inhibition zone measured on four sides of the sample and description of the degree of growth underneath the sample.

Efficacy was assessed with a Zone of Inhibition assay against *Aspergillus niger* ATCC #6275. Petri plates containing Sabouraud Dextrose Agar were inoculated with 0.5 ml of  $1 \times 10^5$  fungal spores/ml. A sample approximately 1 X 1 inch is placed in the center of the plate. The agar plate was incubated for 1-7 days at 25° C. The final data is the average of the inhibition zone measured on four sides of the sample and description of the degree of growth underneath the sample.



5           The percentage of wet pickup employed was between about 50-70 percent, so  
that the actual weight of the fluoro-carbon-containing component was about 1-1.4 percent  
by weight of the treating composition.

Invention  
Example 2

10           This example was prepared as in Example 1, except that 1.0% of Zinc Omadine  
fps dispersion (from Arch Chemical) was used in place of the RC5000.

Invention  
Example 3

15           This example was prepared as in Example 1 except that the chemical bath  
contained:

1.25% Unidyne TG-993,

1.0% Repearl F8025

20           1.0% of Zinc Omadine fps dispersion, and

0.25% of Arkophob DAN.

Invention  
Example 4

25           This example was prepared as in Example 3 except that no Arkophob® DAN  
was used.

Invention  
Example 5

30           This example was prepared as in Example 4 except that the chemical bath  
contained:

1.25% Unidyne TG-992,

1.0% Repearl F7105, and



5 1.0% Zinc Omadine fps dispersion.

Comparative  
Example 6

10 Same as example 1 except that neither hydrophobic crosslinker Arkophob® DAN  
nor antimicrobial agent RC5000 was used.

Comparative  
Example 7

15 The same procedure and materials were employed as in example 1 except that  
no RC5000 was used.

Comparative  
Example 8

20 Same as Example 1 except that Repearl F8025 was used in place of Unidyne  
TG-993.

Example 9  
Prior Art

25 The fabric is Crypton™ 404 obtained from C.F. Stinson and Company of  
Rochester Hills, Michigan (a distributor of Hi-Tex Crypton brand textiles). This is a  
commercial product based upon the teachings of U. S. Patent Nos. 6,024,823,  
6,492,001 B1, and 5,565,265 to Rubin et al.

30 Example 10  
Prior Art

Same as Example 10 except that Crypton™ 61238 from C.F. Stinson was used  
instead of Crypton™ 404.

Example 11  
Control

This example used a textile treated only with water, to test the baseline antimicrobial activity.

5 Table 1: *Results*

Examples	Ex. 1	Ex. 2	Ex. 3	Ex. 4	Ex. 5	Ex. 6	Ex. 7	Ex. 8	Ex. 9	Ex. 10	Ex. 11
Oil Repellence	6	6	6	6	6	6	6	7	6	2	X
Water repellence	3	3	8	5	4	0*	3	10	9	6	X
Corn oil release	4.5	5.0	4.5	4.5	4.5	5.0	5.0	4.5	2.0	2.0	X
Tanning oil release	4.5	4.5	4.5	3.5	3.5	5.0	4.5	1.0	1.0	1.0	1.0

Inhibition (mm) // Growth Under (yes/no)

<i>S. aureus</i>	0//no	4.3//no	0.5//no	6.5//no	2.6//no	0//yes	0//yes	0//yes	0//yes	3//no	0//yes
<i>K. pneumoniae</i>	1.8//no	8.3//no	3.8//no	7.8//no	5.8//no	0//yes	0//yes	0//yes	0//yes	1//no	0//yes
<i>A. niger</i>	0//yes	0//no	0//no	5.8//no	2//no	0//yes	0//yes	0//yes	0//yes	x	0//yes

Note. \* failed to repel water.



5 10.0% Zonyl 7040,  
0.25% Aerotex M3, and  
0.60% Ultrafresh DM-25.

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Table 2: Results

Examples	Ex. 12	Ex. 13	Ex. 14	Ex. 15
Oil Repellence	7	6	6	7
Water epellence	5	3	10	10
Corn oil release	4.0	4.0	2.0	1.5
Burned motor oil release	4.0	4.0	2.0	1.0
	Inhibition (mm) //	Growth	Under	(yes/no)
S. aureus	0//no	8.8//no	0//yes	0//yes
K. pneumoniae	0//no	10.3//no	0//yes	0//yes

Abrasion Testing

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Fabrics from examples 12 to 17, as indicated below, were abraded 5000 cycles using a Martindale abrasion Tester by ASTM D 4966-98@12 kpa. Repellency and release properties were measured again in the same manner as un-abraded samples. Results are listed in Table 2A, below.

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Table 2A: After Abrasion Results

Examples	Ex. 12	Ex. 13	Ex. 14	Ex. 15
Oil Repellence	4	3	2	4
Water Repellence	2	2	4	7
				10
Corn oil release	5.0	4.5	3.5	1.5
Burned motor oil release	4.5	4.0	2.0	1.5

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It was found that the compositions of the invention when applied to a textile result in better repellency at lower concentrations on the fabric. Lesser amounts of treating agent than that which is known in the art may be used in the practice of the invention. Further, superior soil release can be obtained by employing compositions of the invention. Less fluorocarbon-containing material was required in the practice of the invention, as compared to prior art compositions.

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It is understood by one of ordinary skill in the art that the present discussion is a description of exemplary embodiments only, and is not intended as limiting the broader aspects of the present invention, which broader aspects are embodied in the exemplary constructions. The invention is shown by example in the appended claims.